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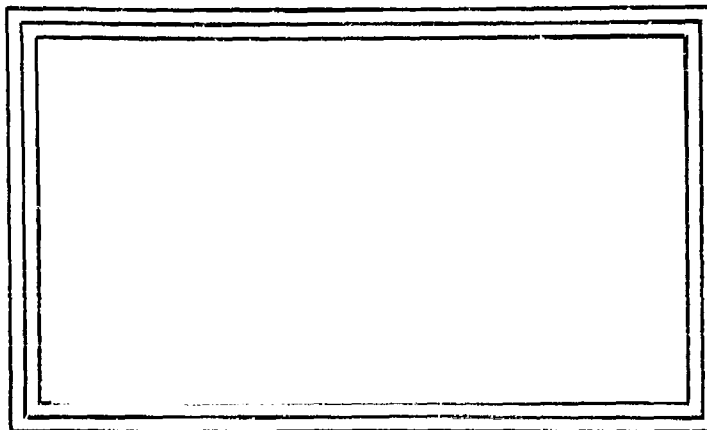


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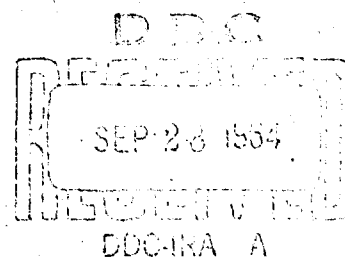
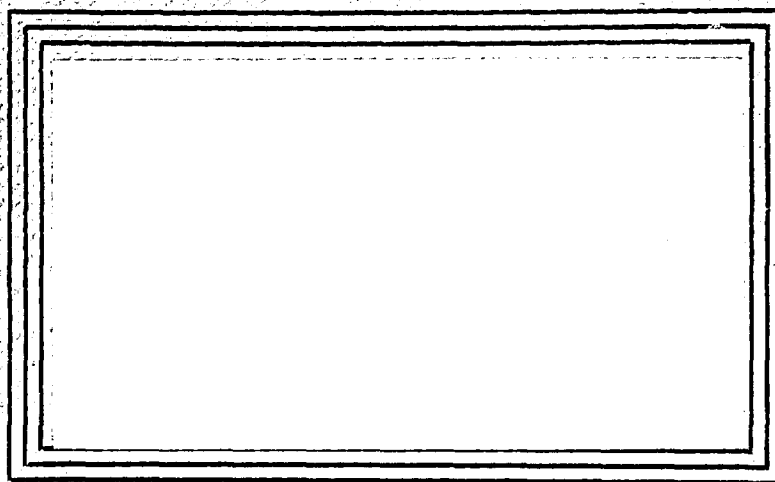


RESEARCH AND DEVELOPMENT DEPARTMENT

U. S. NAVAL AMMUNITION DEPOT - CRANE, INDIANA

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RESEARCH AND DEVELOPMENT DEPARTMENT

U. S. NAVAL AMMUNITION DEPOT - CRANE, INDIANA



RDTR No. 43
10 July 1964

COLORED FLARE INGREDIENT
SYNTHESIS PROGRAM

U. S. NAVAL AMMUNITION DEPOT
Crane, Indiana

RDTR No. 43
10 July 1964

COLORED FLARE INGREDIENT SYNTHESIS PROGRAM

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TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT.	ii
1. PURPOSE	1
2. BACKGROUND.	1,
3. PROGRAM THEORETICAL CONSIDERATIONS.	1
a. Application	1
b. Idealistic Compounds.	2
c. Compound Form	3
d. Compound Composition.	6
e. Potential Synthesis Routes.	7
(1) Acid-Base	7
(2) Alcohol-Metal	7
(3) Organometallics	8
(4) Oxygen and Halogen Attachment	8
(5) Coordination Compounds.	9
f. Compound Properties	10
g. Synthesis Route Selection	14
h. Selection of Reactants.	15
i. Program Synopsis.	20
4. PROGRAM INVESTIGATIONS.	21
a. General Discussion.	21
b. Red Flare Compounds	22
c. Green Flare Compound.	25
d. Work Review	25
5. FUTURE PROGRAM PLANS.	26
6. SUMMARY	27
REFERENCES.	29-31
DISTRIBUTION LIST	32

ABSTRACT

A program is described for the synthesis of compounds whose potential use is for production of colored flames. Theoretical considerations which apply to the conduct of the program are discussed. The properties which an ideal compound would possess are listed. Emphasis is placed upon the preparation of stable, solid, infusible, coordination compounds. The goal is that each molecule of the compound will contain (a) a metal selected for its emission properties when thermally excited, (b) a fuel and oxidant which, when initiated, will react with one another to provide the necessary thermal energy for the metal excitation and sometimes (c) a halogen source which, under specific conditions, will enhance the metal emission. Some guidance is given to describe the variables affecting the selection of the reactants.

A compound prepared under the program and tentatively identified as tris(glycine)strontium(II)perchlorate is mentioned to show that stable compounds of the type described can be formed. The potential formation and usefulness of lithium perchlorate and copper nitrate complexes with glycine are discussed. Effort in the future will be directed primarily toward the preparation of compounds for potential use in green flame production.

COLORED FLARE INGREDIENT SYNTHESIS PROGRAM1. PURPOSE

This presentation is intended to describe a support study program as it relates to the synthesis of new and more efficient compounds whose potential use is principally for the production of colored flames.

2. BACKGROUND

The need for a wider selection of ingredients from which compositions may be formulated to produce colored flames has been recognized long ago. Improved and more efficient compounds are needed. Ellern¹ recognized these deficiencies by specifically listing lack of choice in available color-creating materials as a problem which besets colored flame production, and by emphasizing the necessity to compromise because the most desirable thermodynamic and combustion properties and desirable behavior of the reactants may not all be attainable in one formula. Although the "lack of choice" problem is not the only problem associated with colored flame production, it is one which can be alleviated by synthesis of new compounds which are constructed on a custom basis to efficiently perform specific tasks. With this background and purpose, a program was initiated for the synthesis of improved pyrotechnic ingredients.

3. PROGRAM THEORETICAL CONSIDERATIONSa. Application

(1) Compounds with a variety of properties can be synthesized for numerous pyrotechnic uses. For purposes of this paper,

discussion will be limited to the synthesis of compounds which are suitable for the production of colored flames.

(2) The program includes not only the synthesis of, but also the search for, ingredients which possess the described attributes. Therefore, the author will appreciate information concerning materials which exhibit a significant number of the beneficial properties.

b. Idealistic Compounds

(1) The theory and attributes associated with the production of colored flames were discussed by Douda². In an effort to comply with the postulates which he set forth, it is concluded that available materials have many undesirable properties which must be accepted in order to utilize a desirable characteristic. Because of this, every formula designed to produce a colored flame is the result of many compromises. It is the intent of this program to make more compounds available from which to select, thereby moderating the necessity to compromise. To do this, it is necessary to know what properties are desirable and what characteristics must be avoided.

(2) Some of the important preferred attributes of a colored flame producing ingredient are:

(a) Optimum elemental arrangement which permits compliance with the seven postulates summarized by Douda². These postulates deal with such parameters of colored flame production as emitter selection, flame operating temperature, ionization buffer,

avoidance and formation of molecules within the flame, chemiluminescence and enhancement effects, emission suppressors and extraneous matter.

(b) Durability, safety and environmental stability: These attributes encompass characteristics such as insensitivity to mechanical and electrostatic forces; stability when subjected to wide ranges of temperature and pressure; general compatibility with other chemicals and materials.

(c) Physical and thermodynamic properties: These include characteristics such as water insolubility, non-toxicity of the reactants and the combustion products, low specific gravity, maximum output of heat per unit volume and agreeable handling properties.

(d) Low cost and abundant supply.

(3) Admittedly, it is impossible to synthesize a compound which will display all of the desired properties. Since, however, available materials do not always perform their function in an efficient manner and frequently have other undesirable properties, there is ample room for improvement. Thus, synthesis of new and more efficient ingredients was chosen as a route to alleviate the "lack of choice" problem.

c. Compound Form

(1) Any compound will exist as a gas, liquid or solid at standard temperature and pressure. Which of these three phases is the best for pyrotechnic use is debatable. Many factors will

influence the final choice. The phase preferred for a particular set of conditions will probably be undesirable if the conditions change. It is with this knowledge and understanding that the form of the compound will be discussed.

(2) In order to create a colored flame it is necessary to raise the temperature of the ingredients to their ignition point. The energy necessary to effect this temperature change is a function of the heat capacity of the ingredients. In addition, if fusion, vaporization or some other transition takes place before ignition, the energy associated with that change will have an effect upon the thermal output of the unit. The heat of vaporization of a compound is usually high in relation to its heat of fusion or heat capacity. All of these heats reduce the amount of energy available as output. Thus, high heat capacity or heat of fusion, and especially high heat of vaporization, cause a large expenditure of energy for temperature change, thereby reducing the amount of energy available for other purposes.

(3) In the light of only those considerations described in the preceding paragraph, solid ingredients may be less desirable than liquids or gases because they usually will require more expenditure of energy to reach a given temperature. In this respect, liquids are less desirable than gases. The need for elevation of the temperature until a gas is formed results from the fact that the color of a flame, as used in pyrotechnics, results from the metallic spectra of gaseous excited atoms, molecules or ions.

(a) During the preceding discussion, solids were described as being undesirable under the assumption that fusion and volatilization would take place prior to decomposition. If decomposition takes place before vaporization, and especially before fusion, the handicap is no longer as severe. Under these conditions, the loss of energy due to heats of fusion and vaporization are no longer a consideration. Only heat capacity which is usually small needs to be considered as it does also in the case of a liquid or gas. Thus, a solid is normally desired which not only has the usual desirable properties described in the Idealistic Compounds section, page 2, but also has the ability to decompose prior to fusion or vaporization.

(b) This is one of the principle reasons why solids have been used extensively in pyrotechnic colored flare formulations. Other reasons are associated with reduced sensitivity to electrical energy, ease of handling, processing, packaging and dispensing, low vapor pressure and ultimately a reduced cost resulting from simpler designs and processing methods.

(c) In some instances, it is beneficial for purposes of manufacture of the pyrotechnic and processing of the ingredients to use a compound which (a) is a solid with low vapor pressure at 75°C, (b) will melt above that temperature but preferably below 100°C, and (c) will decompose at a temperature well above 100°C without undergoing vaporization. Such a compound may be useful for application of the casting technique to pyrotechnic manufacture. A

comprehensive discussion of pyrotechnic melt casting is presented in a report by Douda³.

(4) As a result of the considerations just described, the synthesis of solid compounds was chosen as a primary goal. Should liquid or gaseous compounds result from the program, their utility will be considered. In addition to possessing the characteristics listed in the Idealistic Compounds section, page 2, emphasis will be directed toward the synthesis of compounds which will decompose without fusion or vaporization.

d. Compound Composition

(1) The preferred attributes of the compound to be synthesized have already been described. To prepare a molecule which will display most of the desirable properties is a formidable problem. Such a molecule must contain one of the metals associated with color production. In addition, it must contain a fuel component and a source of available oxygen. In certain instances when halogens are required for enhancement, the molecules must contain the appropriate halogen. All of these must be assembled into the molecule in proportions suitable for the production of a colored flame. Stoichiometric balance between fuel and oxidant is generally desirable but frequently is adjusted to obtain variations in the combustion rate.

(2) Molecules, of the type described, can be synthesized by several routes and with a variety of properties. The resultant molecules will be dependent on the manner in which the elements and

radicals are joined together.

e. Potential Synthesis Routes

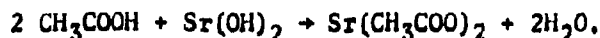
Of the innumerable routes available, those associated with metal-organic chemistry were chosen. This chemical discipline brings together the metal required for color production and an organic component. Because the theory and processes are documented to a large extent, only a brief description of representative reactions have been included in the form of a review.

(1) Acid-Base

Like inorganic acids, organic acids undergo neutralization and salt formation with bases.

For example:

acetic acid + strontium hydroxide + strontium acetate + water



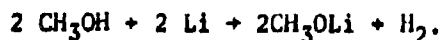
The characteristic feature of this group of compounds is the attachment of the metal through the oxygen of the carboxy group, e.g. $\text{R}-\overset{\text{O}}{\underset{\text{O}}{\text{C}}}-\text{M}$. In an analogous manner, the metal-phenol salts are considered in the acid-base category. Books by Moeller⁴, Richter⁵ and Sienko and Plane⁶ are suggested for additional reading.

(2) Alcohol-Metal

Metals above hydrogen in the electrochemical series react with alcohols by replacing the hydrogen atom on the oxygen.

For example:

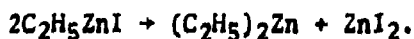
methyl alcohol + metallic lithium + lithium methoxide + hydrogen



The characteristic feature of this group of compounds is the attachment of the metal through the oxygen of the alkoxy group, e.g. R-O-M. Because most of these compounds are subject to hydrolysis, the alkoxides must be considered as very unstable in the presence of water. Fieser^{7,8} and other authors⁹ are suggested for additional reading. The production of alkali metal alkoxides is described by Schechter¹⁰.

(3) Organometallics

Organometallic compounds are characterized by the formation of a bond between the metal atom and one or more carbon atoms, e.g. R-C-M. These compounds are often unstable in the presence of water or air. Sodium ethyl, C_2H_5Na , for example, ignites spontaneously in air as do dimethyl and diethyl zinc. Diethyl zinc¹¹ may be prepared by:



The product is distilled in an atmosphere of carbon dioxide (b.p. $118^\circ C$, yield 81-84%). Extensive treatment of the subject of organometallic compounds may be found in books by Zeiss¹², Kaufman¹³ and other authors⁹. The review by Balueva and Ioffe¹⁴ includes information concerning organic compounds of strontium and barium.

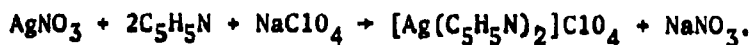
(4) Oxygen and Halogen Attachment

During discussion of the desired makeup of the compound, it was stated that a fuel component is required. This requirement is satisfied by an organic segment to which the metal is

attached. That union is completed using the acid-base, alcohol-metal or organometallic synthesis route. Another necessary component of the molecule is the available oxygen source or the halogen supply. These may be incorporated into the molecule through nitration or halogenation of the organic segment. In some instances, the organic component may be both nitrated and halogenated. An example of such a compound is sodium-3-chloro-4-nitrophenoxide. The perchloro (ClO_3) group cannot be added to an organic structure in a manner analogous to the addition of the nitro (NO_2) group. When the advantages of a perchlorate are desired, this group can be incorporated into the molecule by formation of a coordination compound. It is emphasized that the presence of a source of available oxygen and a fuel component within the same molecule is often sufficient to promote violent decomposition upon ignition.

(5) Coordination Compounds

(a) In 1893 Alfred Werner described the structure and properties of an entirely new field of compounds. These compounds, sometimes called Werner complexes, are better known as coordination compounds. They are compounds formed by combinations of apparently saturated materials, each of which is capable of independent existence. For example, dipyridinesilver(I)perchlorate is prepared¹⁵ by:



This example illustrates not only the union of a metal with an

organic compound but also the inclusion of an oxygen source by means of the perchlorate anion.

(b) The book by Bailar¹⁶ describes the chemistry of coordination compounds and Ballhausen¹⁷ introduces the crystal or ligand field theory approach to the discussion of inorganic complexes. For additional information concerning these compounds, the reader is referred to works by Pauling¹⁸, Schumacher¹⁹ and others^{20,21,22}.

f. Compound Properties

(1) Because of the ionic nature of the salts formed from the acid-base reaction, the solid compounds will generally be infusible or fusible with difficulty. In one respect, this property is considered to be a decided advantage when applied to pyrotechnic uses. If decomposition takes place without fusion, the heats of fusion and vaporization, which are usually large in relation to heat capacity, do not have to be considered. More net heat, therefore, is available from the pyrotechnic than there would have been if fusion had taken place during the process. Another desirable feature of the acid-base reaction is its high yield and speed of the reaction. Because of its ionic nature, the reaction usually takes place instantaneously and with yields approaching 100 percent. From a practical and economic standpoint, these reaction characteristics are most beneficial.

(2) It was previously mentioned that the metal-alkoxide and organometallic compounds are often water reactive and in some

instances air reactive. The lower homologues of a series are frequently liquids. For conventional pyrotechnic purposes, these properties are undesirable. They do, however, warrant consideration along with other materials in programs associated with pyrotechnic applications of water and air reactive materials. In contrast to compounds of an ionic nature, the synthesis of the metal-alkoxides and organometallic compounds proceeds in a manner characteristic of most organic reactions. The yield is frequently low and the reaction takes place over an extended period of time. The air and water reactivity, low yield and extended reaction time account for generally higher unit cost in comparison to other compounds.

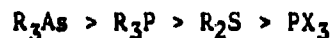
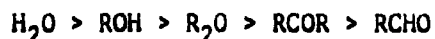
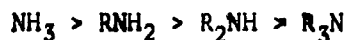
(3) With respect to the properties of coordination compounds, Moeller²³ states that such substances vary widely in their inherent stabilities and other properties. Some appear to exist only in crystal lattices and undergo decomposition into their components when these lattices are disrupted by dissolution or other treatment. Others retain their identities as molecules or ions when dissolved and can be recovered from solution as such. With some, the physical and chemical properties appear to be essentially those of the components; with others, they are completely different. In some, the binding is ionic in character; in others, it is covalent. Because of the potential for a large variety of properties, it becomes more probable that compounds of this type can be of benefit to pyrotechnics.

(4) The idea to use coordination compounds for colored flame production is not new. Ellern²⁴ credits Smith and Koch with proposing, as early as 1935, the use of the alkaline earth amines for colored flame production. Diehl and Wharton²⁵ describe ethylenediamine addition compounds of metal perchlorates and state, "the combination in a single molecule of a combustible, organic component, a potentially powerful oxidant, and a flame colouring agent should give to such compounds some startling properties". Unfortunately, the strontium and barium compounds described by Smith and Koch have, in accordance to Ellern, a dissociation pressure which is uncomfortably large. However, the compounds reportedly do possess the desirable property of being non-hygroscopic. The strontium and barium compounds described by Diehl and Wharton have more tolerable dissociation pressures but are undesirable because of their deliquescent nature.

(5) The group of coordination compounds which contain an oxidant component in the molecule have in common the potential ability to decompose with violence. Diehl and Wharton²⁵ recognized this property in tetrakis(ethylenediamine)strontium(II)perchlorate and tetrakis(ethylenediamine)barium(II)perchlorate. Ellern and Olander²⁶ discuss the explosion of trihydrazinenickel(II)nitrate and Tomlinson²⁷ et. al. describe the explosive properties of metal amines. It was recently reported²⁸ that an explosion occurred during an attempt to prepare cis-dichlorobis(1,2-diaminepropane)chromium(III)perchlorate. These specific examples are cited to

emphasize the potential hazard associated with some compounds of this group. All compounds of this type do not possess this property. Ellern and Olander²⁶ set forth excellent advice concerning this hazard when they stated, "----any of these compounds should be prepared, handled, and stored as if they were initiating explosives until proved otherwise by repeated preparation in an identical manner and by physical tests commonly used for the testing of explosives". Safety precautions applicable to compounds of this type are discussed by McGill²⁹.

(6) In order to minimize the hazard, it would be beneficial if stability could be predicted. Synthesis could then be performed with more confidence and more assurance that a usable compound would result. Although no hard and fast rules are available, Moeller³⁰ listed trends in stabilities as related to the coordinated groups as follows:



where: R = alkyl or aryl radical
X = halide group.

In their approximate sequence of increasing pyrochemical activity, Ellern²⁴ listed coordinating groups of fuel character such as urea, ammonia, ethylenediamine, hydroxylamine and hydrazine. Nitrites,

nitrates, halates and perhalates are listed in the order of their increasing sensitizing action. In addition to the influences just described, the nature of the metal ion must also be considered. The most stable complexes, according to Moeller³⁰, result from cations derived from the transition metals and metals immediately following the transition elements, although the formation of coordination compounds is by no means limited to these materials. The selection of the coordinating group and the metal cation is therefore extremely important because of influences on stability and because these factors also cause variation in pyrotechnical performance.

g. Synthesis Route Selection

(1) All of the potential synthesis routes permit the synthesis of compounds which contain a halogen source, a fuel and oxidant component and a metal for use as a color donor. Alkoxide and organometallic compound syntheses have been described as being less desirable than the other methods from an economic standpoint. In addition, these routes do not allow the introduction of the oxygen supply as a perchlorate. The ability to use perchlorates is an advantage because they contain more oxygen than nitrates and they contain a halogen which is often useful for color enhancement.

(2) The acid-base method is similar to the alkoxides and organometallic routes in the sense that a perchlorate cannot be introduced. The acid-base method does have a decided economic advantage. When a halogen is not required and when sufficient nitrates

can be introduced into the molecule to provide an adequate supply of oxygen without creating an extremely unstable compound, the acid-base route may be particularly useful as a synthesis route.

(3) The route that provides the most versatility is that which leads to the formation of coordination compounds. This mechanism permits the union of an electrically neutral inorganic salt with an organic component. The metal intended to provide the color is the cation of the inorganic salt. The anion may be a halate, perchlorate, nitrate, nitrite, or halide. This is a list of the useful anions. Other anions such as sulfates and phosphates could also be used but are not recommended because of their color suppression capabilities. A wide selection of organic compounds are available or can be synthesized for attachment to the inorganic component. A coordinating ligand containing an element or radical with an unshared pair of electrons must be a part of the organic segment. In addition, as in the case of the reactants used in the other synthesis methods, the organic component may be nitrated or halogenated or both. Because of the great variety of compounds which can be prepared using this mechanism, it was decided to make use of this technique to make new compounds for use in pyrotechnics. In addition, this method has the advantage of generally high yield and low cost.

h. Selection of Reactants

(1) The color associated with a given metal is described by Douda². In most instances only a very few metals are suitable for

a given color. For example, if green is the desired color, usable metals are copper, boron, thallium or barium. From this point, the choice narrows even further when characteristics such as toxicity, cost and preferred wavelength of the emission are considered. Proceeding under the assumption that coordination compounds are the goal, additional consideration must be given to the coordination number of the metal and its ability to coordinate and form a stable compound. The latter characteristics were presented earlier during discussion of the generic properties of coordination compounds. It is unfortunate that the elements that produce the strongest color emission are also the metals which usually are least desirable for coordination purposes. The coordination number is also an important consideration. If the coordination number is large, several organic molecules will coordinate with one mole of the metal. This causes a decrease in the ratio between the amount of metal present and the remainder of the molecule. If the amount of metal is not maintained at a maximum the color emitted will not be sufficiently pure for practical use. In the final analysis, it must be realized that it is the metal from which the color is derived and not from the other components of the molecule. Therefore, the metal content must be maintained as high as possible.

(2) The selection of the organic component also involves many considerations. First, it must contain coordinating ligands. These ligands should preferably be those which form stable compounds with the metal. Relative stability of compounds formed with various

ligands was described previously. Secondly, the organic component should be of low molecular weight. If the molecular weight is low and if the coordination number is also low, the metal content will be high. High metal content has already been shown to be desirable. Low molecular weight is beneficial from another standpoint. It means that less oxidant will be required to oxidize the material. This also aids in keeping the metal content high. Next, the organic component should be saturated and preferably should be a straight chain paraffin. Such compounds burn easier, cleaner and with less smoke formation. Gaydon³¹ discusses the tendency of various fuels to smoke. The aryl compounds are unsaturated and create a large quantity of smoke. For this reason, all compounds containing the benzene ring probably will be less than satisfactory in compounds designed for color production. The inability to use this group is a decided disadvantage because the chemist will not be able to make use of the properties of the ring which permit nitration, halogenation and multiple substitution. Thus potential for considerable versatility will be lost.

(3) The previous discussion of the fuel assumes a predominately carbon substance. Many organic molecules contain elements such as sulfur, phosphorus, boron and silicon. In many instances, these elements are undesirable because of their ability to suppress the metal radiation. This effect is discussed in a report by Douda². Many fuels can contain a large amount of nitrogen in the molecule. This can be a desirable condition from several aspects.

First, the nitrogen in the molecules usually goes off as molecular nitrogen when the molecule decomposes. In oxygen deficient flames, nitrogen is not luminous in the same sense that carbon becomes luminous. This is desirable from a color production standpoint. In addition, the gas assists in the formation of a larger flame plume. This is beneficial from a visibility aspect. Rausch³² recently obtained a patent for lithium perchlorate-hydrazine which is a coordination compound with high nitrogen content.

(4) Although it is desirable to keep the carbon content low in order to keep oxygen requirements at a minimum, the complete absence of carbon can also cause a problem. For example, hydrazine, hydroxylamine and ammonia are excellent from a nitrogen content and coordination standpoint. However, they can form unstable compounds. Thus, compounds such as urea, semicarbazide or glycine may prove to be a satisfactory compromise between low carbon, minimum oxygen requirement and stability. Glycine may have another advantage. The glycine anion is similar to the acetate anion. The glycine anion is also known as the amino-acetate ion. Dean³³ credits the acetate anion with enhancement of barium and strontium emission. Because glycine is so similar, it also may enhance certain metal emissions. If enhancement is not in evidence, it is probable that the glycine anion will not suppress the emission as would be the case with many other anions.

(5) In military pyrotechnics, flames of high luminous intensity are usually required to facilitate observation at great distances.

When a carbonaceous organic fuel is burned, sufficient intensity does not usually result. This problem is often overcome by the addition of magnesium metal to the formula. The burning magnesium liberates a large amount of luminous energy which provides the required intensity along with considerable thermal energy. Organic fuels are not very efficient in this respect. It is, therefore, necessary to recognize this disadvantage when plans are made to use organic materials as a fuel.

(6) Up to this point, the criteria which enter into the selection of the metal and organic component were discussed. The halogen and oxygen components remain for discussion. Because they are often combined in the form of a perchlorate salt such as strontium perchlorate, these two components will be discussed together. When a halogen is required, it is convenient to introduce the halogen in the form of the perchlorate salt of the metal being used for the color emission. Several advantages accrue from this action. The metal, halogen and oxygen are introduced in an efficient form. That is, no extraneous material is carried along with the necessary ingredients. Next, the halogen is present in stoichiometric quantity and is located in proximity to the metal with which it ultimately must unite. This assumes that the halogen is other than fluorine and that the perchlorate anion will not prevent the coordination compound formation. The perchlorate anion, for example, is often associated with the prevention of the coordination reaction. Moeller³⁴ discussed this phenomenon in his treatise. When fluorine

is required, introduction can best be accomplished by fluorination of the organic component usually by substitution of fluorine for hydrogen. Such fluorine becomes a strong oxidizing agent when it is liberated in the flame. An example of a fluorinated organo metallic compound is perfluoro-n-propyl-lithium whose synthesis and stability is described by Beel et.al.³⁵. The other halogens can also be attached to the molecule in the manner described for fluorine.

(7) When the absence of a halogen is required, the choice of oxygen supply will usually lead to the nitro or nitrate group. In this respect, coordination compounds can be formed between an unsubstituted organic molecule and the nitrate salt of the metal being used to provide the color. An organic molecule containing one or more nitro groups may also be used to form the coordination compound with the metal. In summary, it has been stated that the necessary oxygen supply can be introduced as a nitro group on the organic component or as a perhalate anion. The halogen can be introduced through the perhalate anion or as a substitution on the organic molecule. Several combinations of these possibilities may be useful not only to provide the minimal supply of either the oxidant or the halogen but also as a means of influencing or modifying the properties of the final compound.

i. Program Synopsis

As a result of the theoretical considerations, the program was consolidated into the following:

- (1) to synthesize compounds for use in the production of colored flames.
- (2) the compounds will be solids from the class known as coordination compounds.
- (3) consideration will be given to fusible compounds suitable for casting as well as infusible compounds.
- (4) a source of available oxygen, a fuel component, a metal for color emission and a halogen (when such is desirable) will be combined in the same molecule.
- (5) characterization and identification will emphasize the determination of data related to stability.

4. PROGRAM INVESTIGATIONS

a. General Discussion

(1) It was mentioned that the use of coordination compounds for colored flame production is not a new idea. Since several knowledgeable persons have suggested their use, and since these compounds never were accepted by pyrotechnicians, it might be concluded that these compounds are unsuitable for this use. Although it is not obvious, the reason for the lack of acceptance appears to be directly related to stability. In each case that a compound or group of compounds in this category was suggested for pyrotechnic use, sensitivity or low dissociation pressure was mentioned as an undesirable property. It is, therefore, hypothesized that if compounds with sufficient stability for safe processing and use were available, these compounds could be used to increase the efficiency of existing

formulations or to improve performance in other respects. Thus, work was started to prepare compounds which, hopefully, would exhibit the necessary stability.

(2) Improved, more efficient ingredients are needed to produce red, green, yellow and sometimes blue flames. In the process of deciding where to start, blue was ruled out because it is used infrequently; yellow because it is not only the color readily produced by sodium but also the color that results from free carbon luminosity. It was decided to start with red because deep pure green is generally considered to be more difficult to make than red. Thus, what was considered to be the easy route was chosen. This choice was made also with the knowledge that strontium and lithium compounds are readily available and are known to impart a strong red color to a flame. It was reasoned that after the feasibility of the idea was proved, work would be concentrated toward preparation of compounds suitable for use in green flames. It is, after all, the green flame production that is the most problematical and, therefore, allows for a considerable amount of improvement.

b. Red Flare Compounds

(1) A coordination compound tentatively identified as tris(glycine)strontium(II)perchlorate³⁶ was synthesized. From this compound many valuable lessons were learned.

(a) Strontium perchlorate salt will form a complex with glycine. This was not predicted with complete confidence because the nature of the strontium atom is not particularly conducive to

coordination and because the perchlorate anion is known frequently to prevent coordination.

(b) The coordinated compound is water soluble but is not appreciably hygroscopic. The apparent ability of glycine to suppress this intrinsic property of strontium perchlorate is noteworthy. Compounds with desirable properties have often been discarded because they were very hygroscopic or deliquescent. This unfavorable property creates many processing and packaging problems. It is suggested that formation of a coordination compound with glycine may overcome the hygroscopicity problem and as a result, make the compound suitable for use.

(c) The compound is infusible. Infusibility is characteristic of all of the amino acids. Thus, if a fusible compound is desired, the use of an organic fuel component other than the amino acid series will undoubtedly be required.

(d) The compound decomposes above 250°C. A low dissociation pressure is no longer a problem at this level. In addition, the compound is relatively insensitive to impact and electrostatic forces. These very desirable features confirm the feasibility of preparing compounds in this class which can be handled and used with reduced hazard.

(e) Because strontium has a coordination number of six, three bidentate glycine molecules coordinate with one strontium perchlorate molecule. In this ratio, there is insufficient oxygen for complete combustion of the fuel component. Since the

coordination number is an inherent characteristic of the metal, when strontium is used, it may be preferable to use fuels which require less oxygen for complete combustion.

(2) As a result of the information gained from the synthesis of tris(glycine)strontium(II)perchlorate and the determination of its properties, it was decided to attempt to prepare the lithium counterpart of the strontium compound. This work is now in progress and should provide additional data. If hygroscopicity is suppressed, and if the coordination number is four or less, and if a stable compound results, the compound should be usable for colored flame production.

(3) The lithium derivative has another potential use which may be even more important. If it is assumed that a stable, non-hygroscopic compound containing two or less molecules of glycine is formed, the compound will contain enough oxygen to sustain combustion. This being the case, it is assumed that lithium chloride will be a major product of combustion. This assumption is made because Marvin and Woolaver³⁷ state that lithium perchlorate decomposes to lithium chloride and oxygen. Lithium chloride is extremely hygroscopic and, because of this property, it is being considered for use as a hygroscopic nucleus for cloud seeding in weather modification studies. Therefore, the compound may be used for the pyrotechnic generation of anhydrous lithium chloride. The principle advantage of such a compound would be that:

(a) all the necessary materials are combined in one molecule, thereby eliminating the need for preparing an equivalent pyrotechnic by mixing together several ingredients, and

(b) the compound would be nonhygroscopic as compared to lithium perchlorate which would be the only efficient substitute. The homogeneity and stoichiometry that might be achieved would also be a decided advantage with respect to the pyrotechnical performance.

c. Green Flare Compound

Work is in progress to form a compound between copper nitrate and glycine. This approach was chosen to determine what performance can be expected from a nitrate compound in contrast to perchlorate. Dependent upon the coordination number for copper, the anticipated compound may or may not contain sufficient oxygen for combustion. The strength of the color from such a compound will be of particular interest because it will be emitted in the presence of an appreciable amount of carbon. Under those conditions, an acceptable color may not result. Fortunately, powdered copper metal can be added to supplement the color in many instances. This action is usually the most successful in the absence of halogens. Such would be the case if this compound were used.

d. Work Review

Tris(glycine)strontium(II)perchlorate has been prepared. From this compound considerable information resulted which can be used to predict the formation of new compounds and their properties.

Efforts are also being directed toward the formation of lithium perchlorate and copper nitrate coordination compounds with glycine.

5. FUTURE PROGRAM PLANS

a. Efforts will continue toward the preparation and characterization of the lithium perchlorate and copper nitrate coordination compounds of glycine. Subsequently, effort will be directed toward the preparation of compounds suitable for green flame production. Included will be the study of the barium perchlorate-glycine coordination compound. This assumes that formation will occur. Since a diluted color is expected, fuels with less carbon will be studied with respect to their use with the barium salt. Chudinova³⁸ reports that no Cl_2 is found below 520-50°C when barium perchlorate decomposes. By implication, this can be interpreted to mean that Cl_2 is found above 520-50°C and, therefore, that some oxide is formed. If this is the case, particular attention must be given to the combustion temperature in order to keep the oxide formation to a minimum. This will not only affect the combustion of compounds which result from this program but also will influence barium oxide formation in conventional systems.

b. Plans also include the preparation of a calcium perchlorate and glycine coordination compound. Such a molecule would be interesting not only from a color production standpoint but also from the standpoint of calcium chloride formation for use in weather modification studies in a manner similar to that previously described

for the lithium compound. Chudinova³⁸ states that when calcium perchlorate decomposes the decomposition products include the oxide, chloride, molecular chlorine and molecular oxygen. In this event, the system described may not be the most efficient way to produce calcium chloride. However, Schumacher³⁹ states that only a trace of calcium oxide forms. Thus, the oxide may not form in sufficient quantity to present a problem.

c. Silver iodate coordination compound formation with a fuel molecule such as glycine may also be of some benefit for the weather modification program. Such a compound would be expected to decompose to silver iodide whose usefulness for seeding clouds is well known. Although the compound would not be expected to produce color, the iodate anion influence on the compound properties may provide a basis to determine its suitability for use with strontium or barium. Synthesis may be difficult, however, because of the low aqueous solubility of the iodate as reported by Keefer and Reiber⁴⁰.

d. As the program progresses and as more compounds are prepared, it is anticipated that definitive information will be gained which can be used to predict the combinations of fuel, oxidant and metal which will be most beneficial for colored flame production. In addition, that information may be useful for predicting other characteristics of the compound, such as stability.

6. SUMMARY

A program was described which requires the synthesis of more efficient compounds which are to be used for colored flame production.

The theoretical considerations which affect the preparation of these compounds were discussed. Work completed and in progress was reviewed. Tris(glycine)strontium(II)perchlorate is described as a product of this program along with lithium perchlorate and copper nitrate complexes whose preparation and characterization is in progress. The future program plans include a concentrated effort toward the preparation of compounds suitable for the production of green colored flames.

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B. E. Douala
10 July 1964 32p.

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2. Flare, Ingredient
3. Compound Coordination
4. Tris(glycine)strontium
5. Glycine
6. Strontium Perchlorate

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